Impact of In Situ and Ex Situ Annealing on Al-Cu Heterogeneous Nanostructures: A Comparative Study

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Background incl. aims
Water-based aluminum batteries are a promising alternative to commonly used lithium batteries due to properties such as low production costs, safety in handling, and high theoretical capacity [1]. However, the formation of an oxide layer prevents achieving the desired electrochemical properties. By annealing Al-Cu multilayer heterogeneous nanostructures, the formation of a dendritic Al2Cu structure enveloped by pure aluminum can be achieved by a eutectic transformation [2]. Techniques such as FIB (focused ion beam) [3] for sample preparation and in-situ TEM analyses enable detailed monitoring of phase transformations and structural dynamics of materials at high temperatures. Research in this area is key to the development of new materials with potential applications in energy, especially in the context of improving performance and efficiency of water-based aluminum batteries. The main objective of this work is to analyze the structural and phase changes of multilayer nanostructures at elevated temperatures through in-situ annealing in TEM. Another goal is to compare the processes occurring in lamellae annealed in-situ with lamellae created from ex-situ annealed material. The results contribute to a deeper understanding of properties of Al-Cu multilayer heterogeneous nanostructures and their potential for applications. Moreover, they validate the experimental in situ methods employed in studying the microstructural processes.

Methods
Al-Cu multilayer heterogeneous nanostructures, consisting of alternating thin layers of Al and Cu nanoparticles, were prepared using magnetron sputtering. The transmission electron microscopy (TEM) and scanning TEM (STEM) with high-angle annular dark field (HAADF) imaging on a Jeol 2200FS was used for the characterization of samples. This was complemented by energy dispersive spectroscopy (EDS), scanning electron microscopy (SEM) analyses and automated orientation and phase mapping (ASTAR). Cross-sectional lamellae were prepared using a focused ion beam (FIB) on a Zeiss Auriga SEM. In situ annealing was conducted within the TEM using a GATAN heating holder.

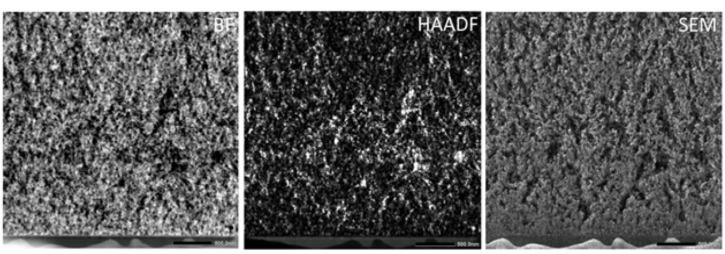
Results

Films of the Al-Cu heterogeneous nanostructures, with thicknesses of 200 nm and 1 µm, were produced. The thin films were annealed in situ to investigate structural changes. Cross-sectional lamellae made from the 1 µm thick films revealed a gradient structure, showing an increasing amount of Al towards the bottom of the specimen. The impact of film thickness on the resultant structures was examined through comparative analysis of in situ annealed lamellae of two different thicknesses and lamellae prepared from ex situ annealed film. Surface modifications, grain size, and the distribution of Al2Cu within Al were mapped using SEM, EDS, and HAADF detectors together with ASTAR imaging techniques.

Conclusion

This study demonstrates the capabilities of in-situ annealing for exploring the structural and phase transformations in Al-Cu multilayer heterogeneous nanostructures. Through characterization techniques, including TEM, STEM, EDS, and ASTAR, a gradient structure with varying aluminum concentration was observed, particularly in multilayer films. The comparisons between in-situ and ex-situ annealed lamellae, highlight the thermal behavior and potential of these nanostructures for advanced material applications, such as in next-generation battery technologies.

Graphic:



Keywords:

Al-Cu multilayer nanostructures, In-situ TEM

Reference:

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